Year: 2012

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Roos, Malgorzata ; Stawarczyk, Bogna

Abstract: OBJECTIVES: This study evaluated and compared Weibull parameters of resin bond strength values using six different general-purpose statistical software packages for two-parameter Weibull distribution. METHODS: Two-hundred human teeth were randomly divided into 4 groups (n=50), prepared and bonded on dentin according to the manufacturers' instructions using the following resin cements: (i) Variolink (VAN, conventional resin cement), (ii) Panavia21 (PAN, conventional resin cement), (iii) RelyX Unicem (RXU, self-adhesive resin cement) and (iv) G-Cem (GCM, self-adhesive resin cement). Subsequently, all specimens were stored in water for 24h at 37°C. Shear bond strength was measured and the data were analyzed using Anderson-Darling goodness-of-fit (MINITAB 16) and two-parameter Weibull statistics with the following statistical software packages: Excel 2011, SPSS 19, MINITAB 16, R 2.12.1, SAS 9.1.3. and STATA 11.2 (p 0.05). Additionally, the three-parameter Weibull was fitted using MNITAB 16. RESULTS: Two-parameter Weibull calculated with MINITAB and STATA can be compared using an omnibus test and using 95% CI. In SAS only 95% CI were directly obtained from the output. R provided no estimates of 95% CI. In both SAS and R the global comparison of the characteristic bond strength among groups is provided by means of the Weibull regression. EXCEL and SPSS provided no default information about 95% CI and no significance test for the comparison of Weibull parameters among the groups. In summary, conventional resin cement VAN showed the highest Weibull modulus and characteristic bond strength. SIGNIFICANCE: There are discrepancies in the Weibull statistics depending on the software package and the estimation method. The information content in the default output provided by the software packages differs to very high extent.

DOI: https://doi.org/10.1016/j.dental.2012.04.013

Originally published at:
Roos, Malgorzata; Stawarczyk, Bogna (2012). Evaluation of bond strength of resin cements using different general-purpose statistical software packages for two-parameter Weibull statistics. Dental Materials, 28(7):e76-e88.
DOI: https://doi.org/10.1016/j.dental.2012.04.013

Journal: Dental Materials

Evaluation of bond strength of resin cements using different general-purpose statistical software packages for two-parameter Weibull statistics

Malgorzata Roos[1], Bogna Stawarczyk[2]

[1]Division of Biostatistics, Institute of Social and Preventive Medicine, University of Zurich, Switzerland

[2]Department of Prosthodontics, Ludwig-Maximilian-University Munich, Germany

Short title: Different packages for Weibull statistics

Corresponding author details:

Malgorzata Roos, PhD
Division of Biostatistics, Institute of Social and Preventive Medicine, University of Zurich, Switzerland
Hirschgraben 84, 8001 Zurich, SWITZERLAND
Tel. +41 44 63 44 648
Fax +41 44 63 44 986
Email: mroos@ifspm.uzh.ch

Abstract

Objectives: This study evaluated and compared Weibull parameters of resin bond strength values using six different general-purpose statistical software packages for two-parameter Weibull distribution. *Methods*: Two-hundred human teeth were randomly divided into 4 groups (n=50), prepared and bonded on dentin according to the manufacturers' instructions using the following resin cements: i) Variolink (VAN, conventional resin cement), ii) Panavia21 (PAN, conventional resin cement), iii) RelyX Unicem (RXU, self-adhesive resin cement) and iv) G-Cem (GCM, self-adhesive resin cement). Subsequently, all specimens were stored in water for 24 h at 37°C. Shear bond strength was measured and the data were analysed using Anderson-Darling goodness-of-fit (MINITAB 16) and two-parameter Weibull statistics with the following statistical software packags: Excel 2011, SPSS 19, MINITAB 16, R 2.12.1, SAS 9.1.3. and STATA 11.2 ($p \leq 0.05$). Additionally, the three-parameter Weibull was fitted using MNITAB 16. *Results*: Two-parameter Weibull calculated with MINITAB and STATA can be compared using an omnibus test and using 95% CI. In SAS only 95% CI were directly obtained from the output. R provided no estimates of 95% CI. In both SAS and R the global comparison of the characteristic bond strength among groups is provided by means of the Weibull regression. EXCEL and SPSS provided no default information about 95% CI and no significance test for the comparison of Weibull parameters among the groups. In summary, conventional resin cement VAN showed the highest Weibull modulus and characteristic bond strength. *Significance*: There are discrepancies in the Weibull statistics depending on the software package and the estimation method. The information content in the default output provided by the software packages differs to very high extent.

Keywords: shear bond strength, dentin, resin cements, three-parameter Weibull, two-parameter Weibull, characteristic bond strength, Weibull modulus, threshold, EXCEL, MINITAB, R, SAS, SPSS, STATA

1. Introduction

In recent years, the demand for non-metallic restorations in dentistry has increased, based on the enhanced aesthetic outcome. Glass-ceramic fixed dental prosthesis (FDPs) must be adhesively cemented, and by using resin cements, the stability and the clinical long-term successes are improved [1-4]. Resin cements not only improve the retention of dental restorations but also exhibit low solubility and microleakage in the oral environment compared with traditional cements, such as glass-ionomer or zinc phosphate cement [5-10]. A multistep pretreatment is required to achieve a compound between the cement and the tooth. Therefore, a multistep adhesive system is needed to remove the smear-layer and additionally create a hybrid layer between the tooth and the cement [11]. Adhesive systems show a high sensitivity for user handling errors which results in significantly lower bond strength values on dentin, based on the fact that the surface is contaminated with saliva or blood or when manufacturers recommendations are not fulfilled [12,13].

These challenges resulted into the development of self-adhesive resin cements, which represent a new system of adhesive bonding on teeth without the necessity of dentin pretreatment. The bonding on dentin without the removal of the smear-layer facilitates the clinical handling and enables the clinician to work more effectively. These resin cements combine the advantages of conventional resin cements, such as adhesive luting to the teeth, reinforcement of the reconstruction, and binding on all different kinds of framework materials, with the advantages of the passive luting agents (simple use without pretreatment of the dentin and less technical sensitivity).

The resin cements are only good, if they have simple application steps. Apart from the chemical composition, handling can impact the bond strength results, mainly the standard deviation and consequently the precision and quality of the adhesion. McCabe and Carrick [14] suggested that less emphasis should be placed on the mean values and standard deviations of the strength when evaluating mechanical properties. Data of mechanical tests of materials may be analysed by a Weibull distribution in which failure probability can be predicted at any level of stress, providing information about the variability of the results and reflecting the structural precision of the materials or bonded assemblies [15-20]. Three- or two-parameter Weibull distributions can be considered. Harper et al. [21] showed that the estimation of the three-parameter Weibull in MINITAB and SAS is possible. However, one should be cautious when using SAS. According to them SPSS and STATA offer just the two-parameter Weibull. Therefore, an example of the three-parameter Weibull estimation was computed exclusively with MINITAB. Anderson-Darling goodness-of-fit estimates were computed to find the best fitting distribution in each resin group. Typical analysis in dentistry considers sample sizes not exceeding 20 per group. According to Abernethy [22] for small samples (at most 20 observations per group) estimation of the three-parameter Weibull is not creditable. Best practice suggested by him is to always use the two-parameter Weibull in such a case. The two-parameter Weibull distributions (with threshold set to 0) is supported by all well-established, general-purpose statistical software packages considered in this investigation, such as MINITAB, R, SAS, SPSS and STATA. When performing a statistical analysis the user generally expects the key statistical results to be the same from different software packages. However, these statistical software packages use different estimation methods for the parameters of the two-parameter Weibull distribution. Typically, the user is not explicitly informed which estimation technique is used by the statistical program. Therefore, the aim of this study was to calculate the two-parameter Weibull statistic with five different general-purpose statistical software packages and to compare the results and the information contained in their output.

The first hypothesis of this study was to test whether all used statistical software packages facilitating the calculation of the two-parameter Weibull statistic showed similar Weibull moduli and characteristic bond strength estimates. The second hypothesis was to test whether similar information content in the default output for the four tested resin cement groups was provided by the five statistical software packages. The third hypothesis was to test whether the four cement groups do not differ with respect to their modulus (m) and characteristic bond strength (s) of the underlying two-parameter Weibull distribution.

2. Material and methods

Two conventional resin cements with self-etch adhesive systems: Variolink II (VAN) and Panavia21 (PAN), and two self-adhesive resin cements namely, RelyX Unicem (RXU) and G-CEM (GCM) were tested in this study (Table 1).

2.1 Specimen preparation

For this investigation, 200 caries-free human molars were collected and directly cleaned. Consequently, all teeth were stored in 0.5% chloramine T solution (Sigma-Aldrich, Seelze, Germany) at room temperature for a maximum of 7 days. Afterwards, they were stored in distilled water for a maximum of 180 days at 5°C until the bond strength tests.

In order to achieve flat surfaces, the buccal surfaces of the teeth were levelled parallel to the tooth axis using a polishing machine (LaboPol-21, Struers, Bellerup, Denmark) with SIC paper P400 (ScanDia, Hagen, Germany). The teeth were then embedded in self-curing acrylic resin (ScandiQuick, ScanDia) and ground finished with carbide polishing paper (P500, ScanDia) until a sufficient bond area of 4x4 mm dentin surface was exposed.

The embedded teeth (N=200, n=50 per cement) were then randomly divided into 4 cement groups (VAN, PAN, RXU, GCM). Prior to the application of the resin cement systems, the dentin test area was ground with the fine polishing paper P500 under water-cooling. While the self-adhesive cements did not require dentin conditioning, the corresponding adhesive systems of the conventional resin cements were applied to the dentin prior to cementation according to the manufacturer instructions. The specimen's stepwise preparation is showed in Figure 1 a–h. Subsequently, the resin cements were activated and placed in an acrylic cylinder (inner diameter: 2.9 mm) that was pressed onto the dentin surface by a holding device (Fig. 1a). In order to achieve homogeneous dispersion of the cement in the acrylic cylinder, a hexagonal steel screw with an outer diameter of 2.8 mm was inserted parallel to the long axis of the acrylic cylinder in its centre (Fig. 1b). A load of 100 g was applied on the screw (Fig. 1c) and the excess cement was removed using microbrushes (Fig. 1d). This procedure allowed resin cement thickness in non-luted state of 0.5 mm in all specimens. VAN, RXU and GCM were dual-polymerised (Fig. 1e) and PAN was chemically polymerised.

2.2 Shear bond strength test

The prepared bond strength specimens (Fig. 1f) were analysed in the Universal Testing Machine (1 mm/min; Zwick/Roell Z010, Zwick, Ulm, Germany). The specimens were positioned in the sample holder of the testing machine with the tooth surface parallel to the chisel-shaped loading piston loading with a width of 1 mm (Fig. 1g) and then loaded until fracture (Fig. 1h). The bond strength (MPa) was calculated using the following formula: Force to failure (N) / Bonding area (mm^2).

2.3 Statistical methods

The cumulative distribution function for the three-parameter Weibull distribution is defined by:

$$G(x) = 1 - \exp\left\{-\left(\frac{x - s_0}{s}\right)^m\right\}$$

where (s) is called scale or characteristic value, (m) indicates the shape or Weibull modulus and (s_0) denotes the threshold (location, minimum life, origin, guaranteed minimum life, shift). G(x) is usually interpreted as the probability of fracture for a test specimen and “x” is the shear bond strength of the specimen tested. If (s_0) is positive, it provides a guaranteed failure free period from 0 to s_0. A non-zero threshold parameter should not be used unless it is anchored in the physics of the failure process. Frequently the two-parameter Weibull [23] is considered for small sample sizes. It is obtained by assuming that the threshold is equal to zero (s_0=0). In such a case the following probability density function

$$g(x) = \frac{m}{s}\left(\frac{x}{s}\right)^{m-1}\exp\left\{-\left(\frac{x}{s}\right)^{m}\right\}$$

is obtained. Frequently the Weibull statistic is computed based on the statistical approach [14,15].

Although the parameters of the underlying Weibull distribution are denoted by s and m their estimates obtained from the analysis of the data are identified by “hats” leading to $\hat{s}$ and $\widehat{m}$ respectively. Note that at least five different parameterisations of the Weibull distribution have been proposed [24].

Least Squares Estimates (LS)

The application of the LS approach to the parameters of the Weibull distribution is justified by the following property of the Weibull cumulative distribution function G(x) for s_0=0 with log denoting the logarithm with basis e:

$$\log(-\log(1 - G(x))) = m\log(x) - m\log(s)$$

In order to compute LS, in each sample group, $\hat{G}$. has to be the estimated for each observation [14,24-26]. After ordering the data from the smallest to the largest value the i^{th} position is considered to be a representative population percentage near to which the i^{th} ordered observation falls. After taking the logarithm of the bond strength

(X-axis) it is plotted against log(-log(1-$\hat{G}_i$)) on the Y-axis and the linear regression fit of the scattered points is computed by means of LS with the sum of squares of the vertical distances being minimised. In other words log(-log(1-$\hat{G}_i$)) is regressed onto log(x). Due to the equation stated above, the estimate of the modulus $\widehat{m}$ of the assumed underlying Weibull distribution is the slope of the linear regression. In order to obtain a value of $\hat{G}_i$ McCabe and Carrick [14] suggested that the specimens within one test group should be ranked by calling the weakest specimen as “rank 1” and the strongest as “rank n”. The probability of failure $\hat{G}_i$ for each specimen from a group containing n specimens is given by

$$\hat{G}_i = R_i/(n+1)$$

where R_i is the ranking number of the specimen. This way of computing $\hat{G}_i$ is frequently called mean rank and can be also found in MINITAB and SPSS under differing names, specifically, Herd-Johnson and Van der Waerden respectively.

Many alternative methods for estimating $\hat{G}_i$ in practical applications were suggested [27]. In MINITAB software, several options such as median rank, modified Kaplan-Meier, and Kaplan-Meier (Hazen) are provided for $\hat{G}_i$ estimation. Conversely, in SPSS Rankit, Tukey and Blom choices are used. Abernethy [22] recommends median rank

$$\hat{G}_i = (R_i - 0.3)/(n+0.4)$$

as the most accurate and therefore considered as best approach to estimate Y-axis plotting positions.

Alternatively, ISO 6872 [25], C 1239 [26], Bergman [28], Khalili and Kromp [29], Steen et al. [30], and ISO 20501 [31] suggest the use of the following formula for $\hat{G}_i$ estimates

$$\hat{G}_i = (R_i - 0.5)/n$$

They are called Hazen's plotting positions by Abernethy [22], modified Kaplan-Meier (Hazen) in MINITAB and Rankit in SPSS. Bergman [28] and Khalili and Kromp [29] show that the Hazen rank leads to the least biased estimates of the modulus whereas the mean rank produces the least acceptable outcome.

Different values for $\hat{G}_i$ lead to different plotting positions for log(-log(1-$\hat{G}_i$)) on the Y-axis. For small sample size, the choice of the formula for $\hat{G}_i$ estimation can lead to different scatter plots and consequently to different LS estimates of both the modulus ($\widehat{m}$) and characteristic bond strength ($\hat{s}$). In this study, we provide the LS estimates for the modulus and characteristic bond strength, estimated according to McCabe and Carrick [14]. While LS median and Hazen ranks were applied when using MINITAB, LS mean and Hazen ranks were utilized by SPSS and by the manual calculation using EXCEL.

Maximum Likelihood Estimates (ML)

The iterative procedure for the ML estimation of the Weibull parameters ($\hat{s}$, $\widehat{m}$), which are "most likely" given on the observed data, is justified by the following formula:

$$Likelihood(m, s|x_1, \ldots, x_1) = \prod_{i=1}^{n} g(x_i) = \prod_{i=1}^{n} \frac{m}{s}\left(\frac{x_i}{s}\right)^{m-1} \exp\left\{-\left(\frac{x_i}{s}\right)^{m}\right\}$$

which holds under the assumption of the independence of the realisations x_i from the Weibull distribution with parameters s and m [15,26,27,32]. ML estimation has attractive mathematical properties for large samples such as consistency, asymptotic normality and asymptotic efficiency [21]. One major advantage of ML is automatic computation of the 95% CIs for the Weibull parameters. Furthermore, they are tighter than those for LS. In addition, when using ML it is not necessary to use any formula for determination of the plotting positions $\hat{G}_i$. Quinn and Quinn [15] mention that the ML estimate for the characteristic strength has negligible bias, but small correction factor is usually applied to correct or "unbias" the Weibull modulus estimate [26,31,33]. We do not apply any unbiasing factors here.

Weibull regression

Weibull regression is a way of analysing the differences in the parameters of the Weibull distribution given the different resin groups. The resin groups have to be coded as covariates by dummy coding (d). The Weibull regression in the accelerated failure time (AFT) metric, which seems to be more frequently used in dental medicine [27], is justified by the following equation:

$$\log(X) = \log(s) + \beta_{AFT} d + \epsilon/m$$

where X obeys the Weibull distribution with parameters s and m and ε is the standard Gumbel (0,1) (smalls extreme value or the weakest link) distribution. For the estimation of the parameters within Weibull regression the Maximum Likelihood methodology is applied.

Goodness-of-fit Anderson-Darling estimates for a wide range of distributions and in particular for the three- and the two-parameter Weibull distributions were computed [22]. This statistic measures how well the data follow a particular distribution. The better the distribution fits the data, the smaller this statistic will be. In order to detect the uniformly best fitting distribution for all four resin groups considered here, ranks were assigned to the Anderson-Darling estimates for each group separately. The distribution leading to the smallest sum of ranks over four resin groups is called the uniformly best fitting one.

The results of the statistical analysis with p-values smaller than 0.05 were considered to be statistically significant whereas those with p-values smaller than 0.1 and larger than 0.05 as tendency. The two-parameter Weibull statistics of bond strength data were analysed using following software types: Excel, SPSS 19, MINITAB 16, R 2.12.1, SAS 9.1.3 and STATA 11.2 (Table 2). The estimation of the three-parameter Weibull was demonstrated with MINITAB. Probability plots for both the two- and three-parameter Weibull for both ML and LS (median rank) estimations were computed in MINITAB. The corresponding program codes used for the estimation are given in Appendix.

3. Results

Figures 2 a, b, c and d show the probability plots for shear bond strength for ML and LS (median rank) estimations for two- and three-parameter Weibull. The data do not fit on a straight line when plotted on transformed two-parameter Weibull axes in Figures 2 a and b. Instead, they show a concave downward curvature. The three-parameter Weibull fit in Figures 2 c and d seems to perform better. The results of the Anderson-Darling goodness-of-fit estimates are provided in Table 3. The three-parameter Weibull distribution seems to fit the data better than the two-parameter one. However, it is not the best fitting distribution for VAN, RXU and GCM groups. The three-parameter log normal distribution provided the uniformly best fit for all four resin groups considered here.

3.1 Three-parameter Weibull

Estimates for the characteristic strength (s), modulus (m) and threshold (s_0) in MINITAB for LS and ML estimation for the three-parameter Weibull distribution are given in Table 4. Due to singularity of the variance-covariance matrix the 95% CI(s_0) for PAN and GCM could not be computed under LS (median rank) and ML estimation techniques respectively. For the same reason the equal characteristic strength, modulus and threshold omnibus tests could not be obtained for both estimation techniques. Although we were able to compute the point estimates of the parameters we are unable to evaluate if there are any differences between them. Interestingly threshold (s_0) was estimated to be ca. 5 MPa.

3.2 Two-parameter Weibull

The outputs of two-parameter Weibull statistics of shear bond strength values calculated using different statistical software packages are summarised in Table 5.

3.2.1 Comparison of the default output provided by the different statistical software packages

EXCEL (LS, mean rank, Hazen rank) estimates were hand computed. Approximate information on the 95% CI for the Weibull modulus but not for the characteristic could be obtained. No tests of the Weibull parameters between resin cements could be considered.

SPSS (LS, mean rank called Van der Waerden, Hazen rank called Rankit) only the absolute estimates could be obtained. SPSS provides the estimates without any omnibus test between the resin cements and without 95% CI for the Weibull parameters.

R (ML) only the Weibull regression approach with homogenous modulus was accessible. The 95% CI for Weibull parameters is lacking and has to be computed by hand. In addition Weibull regression to check the differences in the characteristic bond strength between the resin cements under assumption of homogenous modulus is possible.

SAS (ML), in each group Weibull parameters can be computed. The 95% CI is provided automatically for both the characteristic bond strength (s) and the Weibull modulus (m). SAS provides the estimates without any omnibus test of the Weibull parameters between the resin cements. However, the 95% CI enable comparisons of the estimates between the different resin cements. In addition Weibull regression approach to check the differences in the characteristic shear bond strength between the resin cements under assumption of homogenous modulus is possible.

In STATA (ML) the computation of the Weibull parameters can be carried out only by the use of the Weibull regression. Two parameterisations of the Weibull regression in proportional hazards (PH) and as accelerated failure time (AFT) metrics are provided [34]. The parameters in one metric can be simply transformed into parameters in the other metric. The command "ancillary" can be used to test if there are differences in the moduli (m) between the groups. Depending on the homogeneity of the Weibull moduli the differences between the characteristic values (s) can be investigated. The 95% CI for modulus was given directly. The 95% CI for s had to be computed by hand.

In MINITAB (LS, median rank, Hazen rank called modified Kaplan-Meier (Hazen) or ML) probability plots (Fig. 2 a and b) were provided. The ML and the LS estimates of the modulus and the characteristic bond strength according to the plotting position median rank together with the corresponding 95% CI were computed for all resin cements separately. The Bartlett`s modified likelihood ratio tests together with the appropriate Bonferroni post-hoc tests were used to decide if there are differences in the Weibull modulus and in the characteristic bond strength parameters for ML, LS (median rank) and LS (Hazen rank) estimates between the tested resin cement groups.

3.2.2 Comparison of the results for the characteristic bond strength and Weibull modulus

The estimates of the characteristic bond strength $\hat{s}$ and modulus $\widehat{m}$ for each resin cement and each estimation technique are summarised in Table 5. Figure 3 compares the histograms of the data with the estimated densities obtained by the ML approach whereas Figure 4 shows the densities of the Weibull distributions given the parameter mean rank (EXCEL, SPSS), median rank (MINITAB) and ML (MINITAB, R, SAS, STATA) estimates in Table 5.

As far as the LS median rank estimation in MINITAB, no differences were found between Weibull moduli (m) of the tested resin cement groups ($p=0.625$). There are differences between the characteristic bond strengths (s) between the resin cements ($p=0.001$) with a strong difference between VAN (13.3 MPa) and GCM (10.7 MPa). The findings for LS, Hazen rank were similar.

As far as the ML estimation in MINITAB, significant differences were found between Weibull moduli (m) of the tested resin cement ($p=0.004$) with strong evidence that the moduli for VAN (3.8) and GCM (2.3) differ. As the Weibull moduli (shapes) of the Weibull distribution between the resin cements differ it is not allowed to compare the characteristic bond strengths between all four resin cements directly. A similar difficulty is known for ANOVA when the comparisons between the means are valid only if the assumption of equal variance in all groups is not violated. Consequently, we repeated the analysis excluding GCM. According to the Bartlett`s modified likelihood ratio test there is no evidence that the moduli between VAN, PAN and RXU differ ($p=0.449$). In addition, there is no evidence that the characteristic bond strength between the VAN, PAN and RXU differs ($p=0.052$).

As far as the ML estimation within the Weibull regression in STATA, significant differences were found between Weibull moduli (m) of the tested resin cement groups with strong evidence that the Weibull moduli for VAN (3.8, $p=0.001$), PAN (3.5, $p=0.007$) and RXU (3.2, $p=0.036$) differ from GCM (2.3). Between the following three resin cements VAN, PAN and RXU no violation of the homogeneity of moduli could be found. Under assumption of homogeneous modulus for the three resin cements an estimate 3.4 with 95% CI (3.0; 3.9) was obtained. In this model differences between the characteristic bond strength for VAN (13.4 MPa) and RXU (11.5 MPa) could be found ($p=0.035$).

4. Discussion

In the three-parameter Weibull distribution the non-zero threshold parameter may be fitted only if there is a physical justification for its existence. The threshold parameter might be necessary if one of the following reasons occurs. First, if there is adhesion strength of about 5 MPa preventing the specimens to fracture for the load less than 5 MPa. Second, if there is a residual stress near the point of failure that has a strengthening effect on the specimens. Third, if weak specimens were eliminated before the testing process. Fourth, more than one source of failure is controlling defects. As far as the first point is considered, the literature showed clearly that the shear bond strength values lower than 5 MPa could be measured [35,36,37]. Second, we do not think that there is a residual stress near the point of failure that has a strengthening effect on the specimens. Third, up to our best knowledge no elimination of the specimens was performed as all 200 teeth were embedded together at the same time and then blindly divided into 4 groups by one person. Thereafter, the specimens were bonded, stored and the shear bond strength was measured. No proof testing was conducted and we are unaware of any accidentally broken specimens during their handling and preparation. As far as the fourth point is considered, we know only that all measurements ended with a fracture. Adhesive, cohesive or mixed failure modes might have occurred. Unfortunately, no photographs of the fractured specimens were taken. Consequently, a fractographical failure analysis is impossible for the data at hand and we are unable to decide if multiple flaws might control the distribution. In a case where we had pictures, we could have applied the methodology suggested by Jakus et al. [38] or Johnson [39] which was successfully utilised in Stawarczyk et al. [40].

An alternative reason for the poor fit provided by the two-parameter Weibull distribution might be that the weakest link Weibull assumption (which is the premise of the Weibull model) is inappropriate for the shear bond strengths. The Anderson-Darling goodness-of-fit estimates in Table 3 suggest that there might be other distributions fitting shear bond strengths better than the Weibull one. The best fitting distributions differ between the resin cements. We found that the three-parameter log normal distribution was uniformly the best one for all four resin cement groups considered here.

Due to larger flexibility when working with the three-parameter Weibull distribution, a non-zero threshold parameter Weibull distribution will always provide a better fit than a zero threshold Weibull which actually is the two-parameter Weibull distribution. Figure 2 and Anderson-Darling goodness-of-fit estimates in Table 3 support this statement.

Ng [41] described the estimation of the three-parameter Weibull distribution while Harper et al. [21] documented modern software issues for 15 software package and ML methods combinations in estimating the three-parameter Weibull distribution. Considerable variability existing in results reported by different statistical packages was shown. They showed that the estimation of the three-parameter Weibull in MINITAB and SAS is possible, however one should be cautious when using SAS. They mentioned that SPSS and STATA do not provide the three-parameter Weibull estimation yet. Consequently, MINITAB was our statistical package of choice to exemplify the three-parameter Weibull estimation.

Interestingly, the comparison of the estimates in Table 5 obtained for the two-parameter Weibull with those for the three-parameter Weibull in Table 4 shows that the estimates for the characteristic strength (s) and modulus (m) change considerably depending on the fact if the threshold (s_0) is included as in three-parameter Weibull or is set to be zero as in two-parameter Weibull. This finding confirms the results reported by Steen et al. [30]. We were surprised that despite of the large sample size difficulties occurred for the estimation of the 95% CI of certain resin groups and no omnibus tests for the equality of the characteristic strength, modulus and threshold between the resin groups could be computed. We were able to disclose several difficulties in fitting the three-parameter Weibull distribution to the data.

Frequently in the dental material investigations small sample sizes (at most 20 observations per group) are provided. In such a case Abernethy [22] recommends the estimation of the two-parameter Weibull distribution instead of the three-parameter one. Consequently, we feel that the comparison of general-purpose statistical packages for fitting the two-parameter Weibull distribution might be even more relevant than the three-parameter Weibull. We feel that it is an important issue to investigate whether comparable results can be found across different statistical packages in the case of the two-parameter Weibull distribution. Consequently, our main concern here is the two-parameter Weibull fit. Although no perfect fit was obtained for our data set we feel it was still useful for testing performance of statistical packages with the two-parameter Weibull distribution.

The choice of the statistical software package and estimation method can lead to differing estimates of the parameters of the underlying two-parameter Weibull distribution. Only the ML approach leads to equal estimates for differing packages. The main reason is that the statistical software packages use different estimation techniques. The LS estimates vary for differing definitions of plotting positions $\hat{G}_i$. When applying LS approach one should be aware that there are two possible regressions, Y on X and X on Y, which might be considered. The usual way provided by the programs to compute LS estimates is to consider Y= log(-log(1-$\hat{G}_i$)) given X=log(x). It corresponds to minimising the vertical distances between the scatter points and the best fit line. On the other hand fitting X given Y corresponds to minimising the horizontal distances. Therefore different estimates of the Weibull parameters are obtained even though a LS approach is used. Abernethy [22] provided the discussion of the properties of both approaches. In statistical packages considered here only regression of Y on X was offered.

One possible drawback of the ML estimation is that the estimates for modulus (m) might be biased. As none of the programs considered here gives an option to automatically correct for the bias of the ML estimates no such unbiasing factors were considered here. However, the ML estimates might be corrected by an additional manual computation utilising unbiasing factors as suggested in Thoman et al. [33], C 1239-07 [26] and ISO 20501 [31]. It is an important issue which was considered in Stawarczyk et al. [42] using corrections suggested by EN 843-5:2006 [43] for computation of 95% CI(m).

Statistical estimation methods are highly influenced by the number of specimens. With the sample size of n=50, the estimate $\widehat{m}$ of the true two-parameter Weibull modulus (m) would range between m/f and mf with large probability of 95%, where the imprecision factor f=1.34 can be computed according to Nelson [27]. For instance when the true modulus of Weibull distribution is equal to 3 in one particular test group, then the estimates $\widehat{m}$ based on 50 specimens of the theoretical modulus m would range between 2.2 and 4 with probability 0.95. In our study there are 50 specimens in each group. The sample size is fairly large for the typical analysis in dentistry. It is large enough for the estimation of both the three- and the two-parameter Weibull distributions to be admissible and should be large enough for the asymptotic properties of the ML estimators [22] to unfold. Consequently, only minor discrepancies between the LS and ML estimates would be expected. The differences between estimates obtained by LS and ML methods for the two-parameter Weibull show that the sample size is not large enough to make both estimation techniques provide identical results. Consequently, we recommend the clear statement of the statistical method (LS vs. ML? If LS which kind of ranks were used?) applied for computation of parameters of the Weibull distribution in publications in order to make them comparable.

The second main finding is that the default outputs obtained from the statistical software packages highly differ with respect to the content of information about the parameters of the underlying two-parameter Weibull distribution.

Excel (following the formula from ISO 6872:2008 [25]) and **SPSS** (Van der Waerden and Rankit) apply both the mean and the Hazen rank LS approach. In their default output no 95% CI for the parameters of the underlying Weibull distribution are provided. Consequently, no statistical tool for comparing the parameters s and m between differing cement groups is provided. In EXCEL the approximate 95% CI(m) can be computed by hand. The corresponding code is given in Appendix.

R obtains the estimates by means of ML. No 95% CI for the parameters are automatically given. R uses a different parameterization of the Weibull distribution to one suggested by McCabe and Carrick [14] and also used in our study. A suitable transformation of the obtained parameters is necessary to have their values in the parameterizations used in the study of McCabe and Carrick [14] and this study. Weibull regression is provided only under assumption of the homogeneity of the moduli. Within this setting the comparison of characteristic bond strengths is possible.

SAS obtains the estimates by means of ML and gives them together with the corresponding 95% CI for s and m. Weibull regression is provided only under assumption of the homogeneity of the moduli. Within this setting the comparison of characteristic bond strengths is possible.

STATA obtains the estimates by the ML approach within the Weibull regression with only 95% CI for m given by default. The 95% CI for s have to be computed by hand. The command “ancillary” gives an option to check the assumption of the homogeneity of moduli within the Weibull regression approach. Moreover, under assumption of the homogeneity of the moduli the comparison of characteristic bond strengths is possible.

MINITAB offers both the median and Hazen rank LS and ML approach. The useful probability plots are obtained by default. The 95% CL for m und s are automatically provided. The Bartlett`s likelihood ratio test gives hints if the assumption of the homogeneity of the modulus between the tested groups is violated. By picking up a subset of the data set the analysis for the test groups with homogeneous modulus can be conducted. For them the comparison of the characteristic bond strength between the groups can be completed by means of the Bartlett`s modified likelihood ratio test. Interestingly the Weibull regression in MINITAB is provided only under assumption of the homogeneity of the moduli. Within this setting the comparison of characteristic bond strengths is possible.

Figure 4 demonstrates that the Weibull densities RXU and GCM obtained by LS median rank in MINITAB, LS mean rank in SPSS and LS mean rank in EXCEL can vary considerably and differs from the density obtained by ML. Whereas Fig. 2a and 3 visualise the fit to the data provided by the ML estimated Weibull distributions Fig. 2b gives the corresponding piece of information given the LS median rank estimation technique in MINITAB.

We found that MINITAB offers the most extensive amount of information that is relevant for the well-grounded Weibull statistic. The interested user can easily (by default) generate very useful probability plots, 95% CI and the Bartlett`s modified likelihood ratio test for the differences between m and s. Both the median and Hazen rank LS and ML estimation methods are offered. Some caution should be paid in case when the assumption of the homogeneity of the moduli between the groups is violated. In such a case we suggest that the analysis in MINITAB should be complemented by the Weibull regression analysis with command “ancillary” in STATA.

The Weibull modulus gives an indication of the precision of the shear bond strength, with higher values indicating narrower distribution of the bond strength. It is highly relevant to find if certain cement groups show higher Weibull moduli that are statistically confirmed to differ. Within the cement groups for which the assumption of the homogeneous modulus is not violated the comparison between the characteristic bond strengths can be approached. Materials with higher characteristic bond strength, which are statistically confirmed to differ, have desired properties.

We found discrepancies in the ability to detect the differences between the Weibull moduli for the four cement groups. LS MINITAB approach was unable to find that the modulus of GCM was smaller than in remaining resin cements in contrast to the ML MINITAB approach. It is well known that the standard errors of the estimates obtained by ML methodology are smaller [22, 29]. This might lead to more sensible detection of the possible differences between Weibull moduli in case of the ML approach. However, as pointed out by an anonymous referee, careful inspection of Table 5 shows the LS estimates to have tighter confidence intervals than the ML estimates. Therefore, the significant difference must be due to the greater range of point estimates for ML and not increased precision of ML. The trend in confidence interval widths goes against the well-known relationship, which is noted in [22]. It is very surprising as the sample size in the resin groups is fairly large. It might be the result of inappropriately fitting the data to a two-parameter model instead of a three-parameter model. Indeed, the 95% CI is narrower for the ML estimation than for the LS one when three-parameter Weibull is considered (Table 4) with exception of the PAN group. It is surprising as the PAN group is the only one where the three-parameter Weibull was found to be the best fitting distribution (Table 3). Presumably, the 95% CI provided by LS in PAN group are not reliable as it is the group where the variance-covariance matrix did not exist for LS estimation. Both ML approaches in MINITAB and STATA indicated that the self-adhesive resin cement GCM showed smaller two-parameter Weibull modulus compared to the remaining resin cements.

For the analysis of the resin cements VAN, PAN and RXU with homogeneous modulus we found differences between the results for the characteristic bond strength provided by the Bartlett`s modified likelihood ratio test for ML approach in MINITAB and the ML approach within Weibull regression in STATA. The Bartlett`s modified likelihood ratio stated that there was only tendency (p=0.052) for the characteristic bond strengths between the resin cements to differ whereas STATA clearly stated that the self-adhesive resin cement RXU had smaller characteristic bond strength than the conventional resin cement VAN combined with the Syntac System. We prefer to discuss the results obtained by the Weibull regression approach in STATA as the Bartlett`s modified likelihood ratio test in MINITAB is only an approximation to the likelihood ratio test.

Consequently, the third main result provided by the Weibull regression in STATA states that conventional resin cement VAN showed higher Weibull modulus (3.8) compared to the self-adhesive resin cement GCM and showed higher characteristic bond strength (13.4 MPa) than RXU among the three resin cements (VAN, PAN, RXU) having homogeneous Weibull modulus. PAN and RXU cements had higher modulus than GCM but cannot be distinguished from each other with respect to the differences in the characteristic bond strength.

The specimens of this study were fabricated under standardised conditions without clinical situation such as absence of saliva or under time pressure in the patient's chair. Therefore, it can be suggested that Weibull moduli for clinical bond strength are lower than those of this in vitro study. Additionally, the used resin cements could have different sensitivity in clinical situation. Consequently, it is possible that the order of ranking of the Weibull moduli would be different in the oral situation than in this study. Therefore, further studies should consider these aspects when studying the bond strength of resin cements.

Our investigation used shear bond strength for the Weibull calculations. The shear bond strength tests are controversially discussed in the dental literature. One study compared the bond strength results of different test methods and stated that shear bond strength tests showed a higher coefficient of variation compared to all other bond strength methods, e.g. tensile bond strength [44]. The differences are related to the fact that the shear bond strength tests have non-uniform stresses generated within the reaction zone, which can have a significant effect on the mode of failure. Therefore, it could be assumed, that the Weibull moduli of bond strength tested using other test methods are higher compared to those of the present study.

5. Conclusions

Within the limitations of this in vitro study, it can be concluded that:

- Two-parameter Weibull estimates calculated with MINITAB and STATA can be compared using an omnibus test and using 95% CI.
- In SAS only 95% CI were directly obtained from the output. R provided no estimates of 95% CI. In both SAS and R the global comparison of the

characteristic bond strength among groups is provided by means of the Weibull regression.

- EXCEL and SPSS provided no default information about 95% CI and no significance test for the comparison of Weibull parameter among groups.
- In summary, conventional resin cement VAN showed the highest two-parameter Weibull modulus and characteristic bond strength.

Acknowledgments

The authors would like to thank the companies for providing the materials.

Tables

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Figures

Fig. 1: Production process of the specimens. A: acrylic cylinder pressed on the the dentin surface B:cement put in the acrylic cylinder C: steel screw put into the acrylic cylinder D: removed of excess cement E: cement luted F: finishes speciment G: speciment in sample´s holder H: test design of shear bond strength.

Fig. 2: Probability plots for shear bond strength for a) two-parameter ML estimations and b) two-parameter LS estimations c) three-parameter ML estimations and d) three-parameter LS estimations**.**

Fig. 3: The histograms of the observed shear bond strength in each resin cement group together with the estimated Weibull densities according to ML.

Fig. 4: Comparison of the estimated Weibull densities in each resin cement group for four different estimation techniques: ML=ML, LS1=mean rank EXCEL, LS2=mean rank SPSS, LS3=median rank MINITAB.

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Table 1. The abbreviations, brands, manufacturers and Batch-No of the tested materials.

	Abbreviations	Resin cement brands	Manufacturers	Batch-No.
Conventional cements	VAN	Variolink II Adhesive System: Syntac classic	Ivoclar Vivadent, Schaan, Liechtenstein	K41833/K39878 J280035/J27820)
	PAN	Panavia21 Adhesive System: ED Primer A/B	Kuraray Dental Co Ltd., Osaka, Japan	408CA
Self-adhesive cements	RXU	RelyX Unicem	3M ESPE, Seefeld, Germany	361930
	GCM	G-Cem	GC Europe, Leuven, Belgium	0801091

Table 2. Information about software used.

Software	Version	Manufacturer
EXCEL	11.6.5	Office Microsoft Excel 2004 for Mac
SPSS	19	SPSS INC, Chicago, IL, USA
MINITAB	16	MINITAB, State College, PA, USA
R	2.12.1	The R Foundation for Statistical Computing
SAS	9.1.3	SAS Institute Inc., Cary, NC, USA
STATA	11.2	StataCorp, College Station, Texas, USA

Table 3. Anderson-Darling goodness-of-fit estimates for two- and three-parameter Weibull, the best fit distribution per group and the three-parameter log normal distribution.

Group	Weibull(2)	Weibull(3)	Best fit per group	Log normal(3)
VAN	0.94	0.54	0.49 Lognormal(3)	0.49
PAN	1.54	0.51	0.51 Weibull(3)	0.64
RXU	3.91	1.31	0.83 Lognormal(3)	0.83
GCM	6.12	1.10	0.51 Loglogistic	0.57

Table 4. LS and ML estimates of the three-parameter Weibull together with 95% CI.

	$\hat{s}$	$\widehat{m}$	Threshold
	Least squares		
VAN	10.1 (4.6; 21. 9)	2.8 (1.1; 6.9)	3.1 (-4.0; 10.3)
PAN	6.2 (5.0; 7.6)	1.5 (1.1; 1.9)	5.6*
RXU	6.1 (4.4; 8.5)	1.8 (1.2; 2.6)	4.8 (3.4; 6.2)
GCM	5.2 (3.7; 7.4)	1.4 (1.0; 1.9)	5.0 (4.1; 5.9)
	Maximum Likelihood		
VAN	9.2 (6.5; 12.9)	2.5 (1.6; 3.9)	4.0 (1.3; 6.6)
PAN	5.8 (4.3; 7.8)	1. 5 (0.9; 2.2)	5.8 (5.0; 6.6)
RXU	5.8 (4.7; 7.3)	1. 6 (1.2; 2.1)	5.1 (4.6; 5.6)
GCM	5.3 (4.1; 6.7)	1.2 (0.9; 1.5)	5.0*

(*) Variance-covariance matrix of estimated parameters did not exist. SE and 95%CI could not be computed. Tests for equal shape, scale and threshold parameters could not be done because the variance-covariance matrix involved in the test statistics was singular.

Table 5. LS and ML Weibull statistic, characteristic bond strength (MPa) and Weibull moduli calculated using different software with 95% CI and p-values provided by the Bartlett's modified likelihood ratio test in MINITAB.

	Least squares					
	Excel		SPSS		MINITAB	
	Mean rank				Median rank	
	$\hat{s}$	$\hat{m}$, 95%CI(m)	$\hat{s}$	$\hat{m}$	$\hat{s}$, 95%CI(s) p=0.001	$\hat{m}$, 95%CI(m) p=0.625
VAN	13.4	3.8 (3.6; 4.0)	13.4	3.8	13.3^{b} (12.3;14.3)	4.1^{A} (3.3;5.0)
PAN	12.4	3.5 (3.2; 3.8)	12.4	3.5	12.2^{ab} (11.3;13.2)	4.0^{A} (3.3;4.8)
RXU	11.5	3.4 (3.0; 3.9)	11.5	3.4	11.3^{ab}(10.4;12.2)	4.1^{A} (3.5;4.8)
GCM	11.1	2.9 (2.4; 3.3)	11.2	2.9	10.7^{a} (9.8;11.7)	3.7^{A} (3.3;4.1)
	Hazen rank					
VAN	13.3	4.1 (3.8; 4.3)	13.3	4.1	13.3^{b} (12.3;14.3)	4.2^{A} (3.4;5.1)
PAN	12.3	3.8 (3.4; 4.1)	12.3	3.8	12.2^{ab} (11.3;13.1)	4.1^{A} (3.4;4.9)
RXU	11.5	3.7 (3.2; 4.1)	11.5	3.7	11.2^{ab} (10.4;12.1)	4.3^{A} (3.7;4.9)
GCM	11.1	3.0 (2.5; 3.5)	11.1	3.0	10.7^{a} (9.8;11.6)	3.8^{A} (3.5;4.3)
	Maximum Likelihood					
	R		SAS, STATA*, MINITAB			
	$\hat{s}$	$\hat{m}$	$\hat{s}$, 95%CI(s) p=0.035		$\hat{m}$, 95%CI(m) p=0.004	
VAN	13.4	3.8	$13.4^{a, z}$ (12.4;14.5)		$3.8^{B,Z}$ (3.0;4.8)	
PAN	12.4	3.5	$12.4^{a, yz}$ (11.3;13.5)		$3.5^{BA,Z}$ (2.8;4.3)	
RXU	11.5	3.2	$11.5^{a, y}$ (10.5;12.7)		$3.2^{AB, Z}$ (2.5;3.9)	
GCM	11.2	2.3	11.2 (9.8;12.8)		$2.3^{A,Y}$(1.9;2.9)	

*In STATA only the 95%CI(m) are obtained directly from the output.
A,B, a,b: Letters indicate differences between the estimates as indicated by the Bartlett's modified likelihood ratio test in MINITAB (characteristic bond strength (s): small letters, modulus (m): capital letters). Note that the differences between s are tested only for groups with homogeneous modulus m.
Z,Y, z,y: Letters indicate differences between the estimates as indicated by the Weibull regression in STATA (characteristic bond strength (s): small letters, modulus (m): capital letters). Note that the differences between s are tested only for groups VAN, PAN and RXU for which the assumption of homogeneous modulus m could not be rejected.

Fig. 1: Production process of the specimens. A: acrylic cylinder pressed on the the dentin surface B:cement put in the acrylic cylinder C: steel screw put into the acrylic cylinder D: removed of excess cement E: cement luted F: finishes speciment G: speciment in sample´s holder H: test design of shear bond strength.

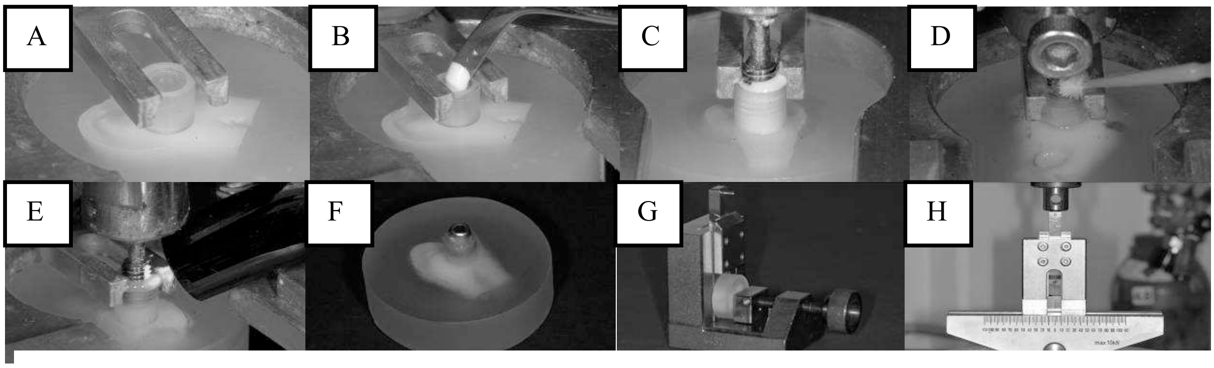


Fig. 2: Probability plots for shear bond strength for a) two-parameter ML estimations and b) two-parameter LS estimations c) three-parameter ML estimations and d) three-parameter LS estimations.

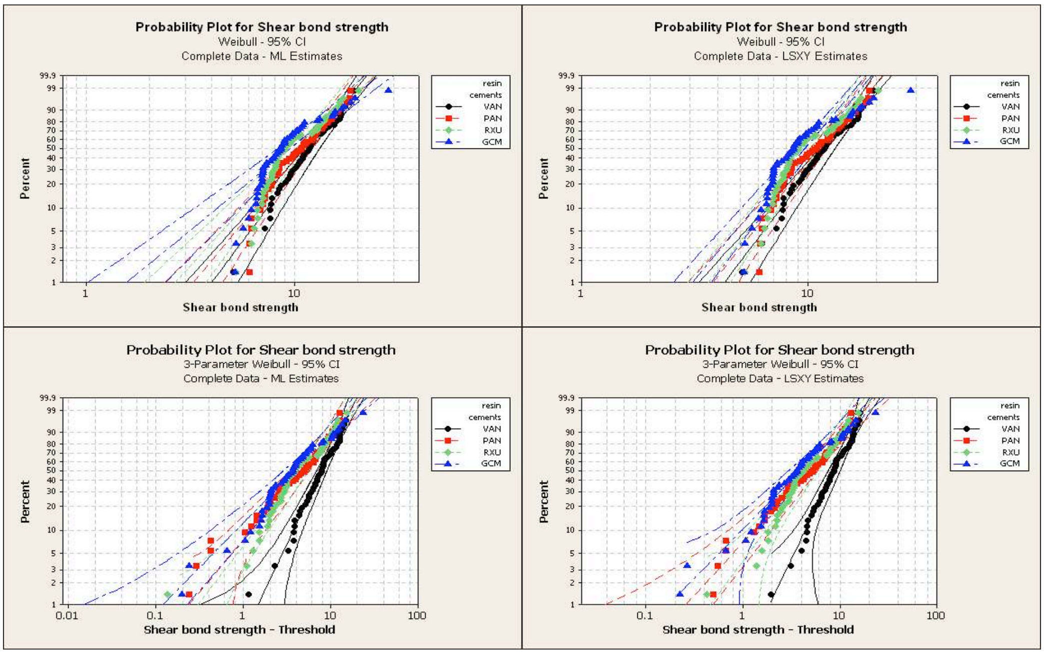


Fig. 3: The histograms of the observed shear bond strength in each resin cement group together with the estimated Weibull densities according to ML.

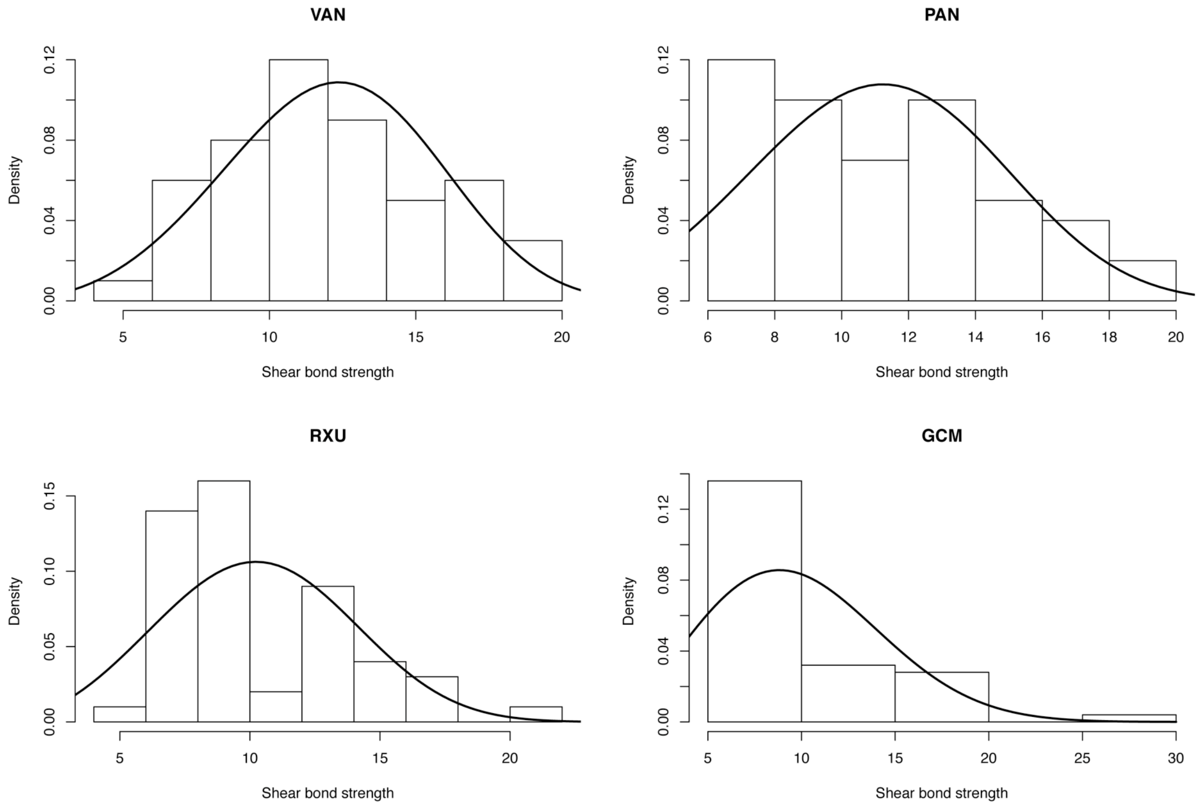
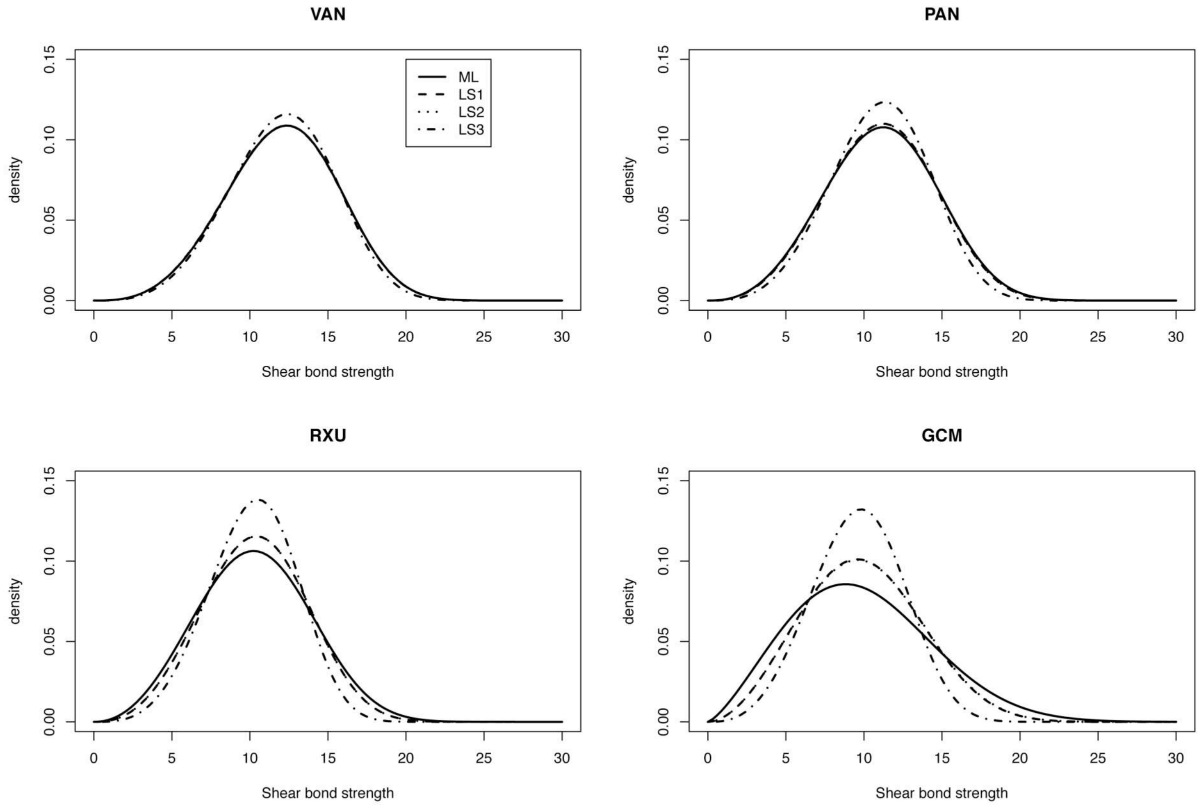


Fig. 4: Comparison of the estimated Weibull densities in each resin cement group for four different estimation techniques: ML=ML, LS1=mean rank EXCEL, LS2=mean rank SPSS, LS3=median rank MINITAB.



Appendix

The self-explaining codes for programs used for the estimation of the two-parameter Weibull distribution are given below:

EXCEL:
Assume that in A the data from one particular resin cement group are already sorted form the smallest to the highest shear bond strength
B contains the corresponding ranks from 1 to 50
C2=B2/51 (HAZEN: C2=(B2-0.5)/50)
D2=LOG(-LOG(1-C2,2.7182818),2.7182818)
E2=LOG(A2,2.7182818)
LS estimation: =RGP($D$2:$D$51;$E$2:$E$51;WAHR)
The value of the slope m is the modulus.
Consider the estimates of the slope m and intercept c to compute the characteristic bond strength s =EXP(-c/m).
95%CI(m): Let sem be the SE of the m estimate, then
Low=m-1.96*sem, Up=m+1.96*sem

Data set used in the statistical software packages:

'resin_cements': group codes 1, 2, 3, 4
'shear_bond_strength': observed shear bond strength values
'fullev': only values 1

SPSS: Path: Analyze/Descriptive Statistics/P-P-Plots/

```
SORT CASES  BY resin_cements.
SPLIT FILE SEPARATE BY resin_cements.

PPLOT
  /VARIABLES=shear_bond_strength
  /NOLOG
  /NOSTANDARDIZE
  /TYPE=P-P
  /FRACTION=VW (Hazen: /FRACTION=RANKIT)
  /TIES=MEAN
  /DIST=WEIBULL.
```

R:

```
library(survival)
#resin1e1 contains “shear_bond_strength” and “fullev” for the first resin group only
wbrege1<-survreg(Surv(shear_bond_strength,fullev,type='right')~1, data=resin1e1,
dist="weibull")
summary(wbrege1)
rwshapee1<-1/wbrege1$scale

rwscalee1<-exp(wbrege1$coefficients)
```

SAS:

```
proc lifereg data=resin;
```

by resin_cements;

model shear_bond_strength*fullev(0) = / dist=Weibull;

run;

STATA:
// initialization of survival analysis
stset shear_bond_strength, failure(fullev==1)
// generation of dummy coded variables for each resin cement group
tabulate resin_cements, generate(dc)
// resin cement group affects both the scale and the shape of the hazard
streg dc1 dc2 dc3, d(weibull) ancillary(dc1 dc2 dc3) time
// The modulus for groups 1, 2 and 3 is assumed to be homogeneous
streg dc1 dc2 if resin_cements<4, d(weibull) time

MINITAB:
The options for the LS estimation have to be changed under:
Tools/ Options/ Individual Graphs/ Probability Plots (Median Rank or modified Kaplan-Meier (Hazen))
Path: Stat/ Reliability-Survival/ Distribution Analysis (Right Censoring)/ Parametric Distribution Analysis/
For ML estimation MLE; instead of LSXY; should be used.
RDIdentification 'shear_bond_strength';

By 'resin_cements';

LSXY;

Ptiles 1 5 10 50;

Allpts.

Ltest 'shear_bond_strength';

By 'resin_cements';

Weibull;

Pplot;

Allpts;

CI;

Brief 2;

LSXY;

Confidence 95.0;

TwoCI;

TESS;

TESL.